

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE****In re PATENT APPLICATION of****JAKOB ET AL.**

Application Serial No.: 09/961,395

Group Art Unit: 1754

Examiner: LISH, P.J.

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Title: GRANULAR SODIUM CARBONATE OBTAINED BY FLUID-BED SPRAY GRANULATION  
AND A PROCESS FOR ITS PRODUCTION**DECLARATION UNDER 37 C.F.R. § 1.132**

Hon. Commissioner of Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

I, Harald Jakob, hereby declare that:

1. I have been employed as a chemist by Degussa AG, the assignee of this application, since 1986. I have been the head of research and development in the area of detergent raw materials since February 2000. As head of research and development for detergent raw materials, I am responsible for all research pertaining to sodium percarbonate.
2. I have received a diploma degree from University Frankfurt/Main, Germany in 1982 and received a doctorate degree (equivalent to a Ph.D.) from University Frankfurt/Main, Germany in 1985.
3. I am a co-inventor of the invention disclosed in U.S. Application No. 09/961,395. I have reviewed the prosecution history of this application, including the Office Actions issued by the examiner and the references relied upon by the examiner.
4. Under my supervision and according to my instruction, the following experimental data was obtained. All experiments were carried out using the same starting materials and conducted at the same process conditions as those disclosed on pages 9-10 of the specification.

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5. The following table lists the TAM value (in  $\mu\text{W/g}$ ) and the dissolving time (in minutes) for various samples run using 0.0-0.5 wt.%  $\text{MgSO}_4$  (corresponding to 0-1000 ppm  $\text{Mg}^{2+}$ ) and 0.0-1.0 wt.%  $\text{SiO}_2$ . The experiments using water glass in an amount of 0.1 wt.% correspond with examples 19-21 set forth in Table 3 on page 13 of the specification.

$\text{SiO}_2$ (wt. %)	$\text{MgSO}_4$					wt. % $\text{MgSO}_4$ ppm Mg
	0	0.07	0.13	0.25	0.5	
	0	125	250	500	1000	
0			18.3		11.1	$\mu\text{W/g}$
			0.7		0.7	min.
		10.4	9.3	6.3	5.5	$\mu\text{W/g}$
0.1		1.4	1.1	0.8	0.7	min.
			7.2	5.6	5.2	$\mu\text{W/g}$
0.25			2.0	2.0	1.1	min.
						$\mu\text{W/g}$
						min.
0.38		7.8	5.4	5.0	4.1	$\mu\text{W/g}$
		2.0	2.6	2.5	3.0	min.
						$\mu\text{W/g}$
0.5	7.6					min.
	2.2					

6. The table demonstrates the surprising and unexpected synergistic results obtained using a magnesium compound in an amount ranging from 125-1000 ppm in conjunction with waterglass in an amount ranging from 0.1-1.0 wt.%. As demonstrated through the data set forth in the table, using 0.1-1.0 wt.% waterglass in combination with 125-1000 ppm magnesium provides products having better TAM values than (a) products prepared using only waterglass, and (b) products prepared using only magnesium sulfate. For instance, a product obtained using both magnesium sulfate and water glass in amounts of 250 ppm magnesium and 0.25 wt.%  $\text{SiO}_2$  has a lower TAM value ( $7.2 \mu\text{W/g}$ ) than (a) a product obtained using only water glass in an amount of 1 wt.%  $\text{SiO}_2$  ( $7.6 \mu\text{W/g}$ ), and (b) a product obtained using only magnesium sulfate in an amount of 1000 ppm ( $11.1 \mu\text{W/g}$ ). The data also demonstrates that small amounts of both  $\text{MgSO}_4$  and  $\text{SiO}_2$  synergistically produce better

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results than using the same amounts of MgSO<sub>4</sub> or SiO<sub>2</sub> alone. For instance, a product obtained using only 0.5 wt % SiO<sub>2</sub> is shown to have a TAM value of 7.8 µW/g. If the reaction mixture is augmented with an additional 0.5 wt % SiO<sub>2</sub>—resulting in a product obtained using 1.0 wt % SiO<sub>2</sub>—the TAM value is only decreased minimally to 7.6 µW/g. However, if the reaction mixture is augmented with an additional 0.5 wt % magnesium sulfate—resulting in a product obtained using 0.5 wt % SiO<sub>2</sub> and 0.5 wt % MgSO<sub>4</sub> (1,000 ppm Mg)—the TAM value drops considerably to 4.1 µW/g. Other synergistic results may be seen throughout the table.

7. The data disclosed in Tables 1 and 2 of U.S. Patent No. 5,714,201 relating to active oxygen (O<sub>a</sub>) retention would not provide sufficient guidance to generate sodium percarbonate samples having low TAM values, such as those set forth in the table above, because O<sub>a</sub> retention and TAM values are unrelated. The O<sub>a</sub> retention is used to determine the storage stability of sodium percarbonate in *detergent formulations* while the TAM value is used to determine the storage stability of sodium percarbonate *in a silo*.

8. Table 5 in U.S. Patent No. 6,413,927 demonstrates that there is no correlation between active oxygen and TAM values. In Table 5, both the TAM values, designated as LKB40, and active oxygen stability in detergent formulations, designated as Avox Recovery, are shown for a number of examples. Comparing the LKB40 data and the Avox Recovery demonstrates that little to no correlation appears to exist between active oxygen stability and TAM values.

9. In view of the lack of correlation between active oxygen and TAM values, I, as a person of skill in the art, would not be motivated to modify the disclosure of U.S. Patent No. 5,560,896 to use a particular amount of magnesium and particular amount of waterglass in view of the teachings of U.S. Patent No. 5,714,201. If the disclosure was modified to use the particular amounts of magnesium and waterglass as claimed in this application, I would not expect that the resulting sodium percarbonate product to be safe for storage in a silo; certainly, I would not expect the resulting sodium percarbonate product to exhibit the synergy and low TAM values set forth in the above chart.

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10. I hereby declare that all statements made herein, of my own knowledge are true and that all statements made on information and belief are believed to be true. Further, I also declare that these statements were made with knowledge that willful false statements, and the like, so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code. I also declare that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

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Harald Jakob

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Date